

RAPID PLASTIC DEFORMATION EFFECT IN SINTERING PROCESS

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ABSTRACT

PLASTIC DEFORMATION EFFECT IN SINTERING PROCESS. The synthesis of a new austenitic alloy, named A-1, has been carried out as a study material for a power-reactor heat exchanger. Powder metallurgy method was utilized in the alloys' synthesis. The alloys elemental components are Fe, Ni, Cr, Si, Mn, C and Ti. Each of the alloying components was first determined in terms of weight-percentages, before actual weighing on the microbalance apparatus. The components were blended together in a special purpose ball-mill for 46 hours. The first samples are in the form of pellets of 25 grams each, obtained by compaction process using a die with a 2.0 cm internal diameter, at a pressure of 8000 psi. After pre-sintering at 1050°C for 24 hours, sintering was carried out at 1250 °C for 6 hours, escorted by rapid plastic deformation treatment that was carried out by forging for every half hour. The microstructure observation shows that the grains form disorder pentagon or more fractional and the high crystal defect or disorder. The distribution observation of elemental were carried out twice by EPMA; the first is as sintering just half hour where the rapid plastic deformation treatment not been carried out yet and the second is at the six hours sintering in which the rapid plastic deformation treatments were carried out.

Keywords: Plastic deformation, rapid and sintering.

ABSTRAK

PENGARUH DEFORMASI PLASTIS CEPAT DALAM PENYINTERAN. Telah dilakukan pembuatan bahan austenitik baru yang dinamakan dengan A-1 sebagai bahan studi untuk penukar panas reaktor daya (PLTN). Pembuatan bahan paduan ini dilakukan dengan teknik metallurgi serbuk. Komponen unsur-unsur paduannya adalah Fe, Ni, Cr, Si, Mn, C, dan Ti. Masing-masing komponen bahan ditentukan persen beratnya dan ditimbang. Campuran unsur-unsur tersebut diaduk dengan ball milling selama 46 jam. Masing-masing sampel awal yang dibuat berupa pelet bermassa 25 gram, diperoleh dengan kompaksi menggunakan die berukuran diameter dalam 2,5 cm, dengan tekanan kompaksi 8000 psi. Setelah diberi perlakuan presinter pada 1050 °C selama 24 jam, sampel disinter pada temperatur sekitar 1250 °C selama 6 jam disertai perlakuan deformasi plastis dengan penempaan yang dilakukan dalam setiap selang setengah jam. Pengamatan struktur mikro dengan mikroskop optik menunjukkan bahwa bentuk butir fasa umumnya berbentuk pecahan segi lima atau lebih yang tidak teratur, dan mengandung jumlah disorder kristal yang tinggi. Pengamatan distribusi unsur dilakukan dua kali dengan EPMA, yaitu pada saat proses sinter baru berlangsung setengah jam saat belum diberi perlakuan deformasi plastis cepat dan pada kedua saat sinter berlangsung enam jam yang diberi perlakuan deformasi plastis cepat.

Kata kunci: Deformasi plastis, cepat, and penyinteran

INTRODUCTION

To build the nuclear power plant (NPP), it is needed to master structure materials manufacture which had to have several advanced properties as well as must be stable with respect to temperature operation, and has a good mechanical properties due to the fact that it needs to be stable at stressed condition ^[1], and resist to high temperature corrosion and also sensitization ^[2]. This paper discussed the austenitic sample synthesis from the powder elements components which also has been discussed in the previous paper ^[1], but with the different sintering method. In the previous paper, after pre-sintering and sintering process, the homogenization process was interrupted by several rapid plastic deformation treatments, meanwhile in this paper the process was inverted, where rapid plastic deformation treatment was carried out during the sintering process. This method was inspired from the last method, where the homogenization process was attended by rapid plastic deformation interruption. In this case, the rapid plastic deformation interruption was carried out every half hour during the sintering process; the rapid plastic deformation treatment was purposed to rise the material's Gibbs free energy ^[3] so that the alloying elements could be alloyed more effectively. It is well known that the rapid plastic deformation will destruct the crystal arrangement to produce plenty of dislocations meanwhile the rise of dislocation will rise the material bulk internal energy, and this is meant that the material's internal energy increases and so that it is in unstable or metastable condition. In thermodynamic term, the increase of internal energy that also named by Gibbs's free energy or inner potential energy, will support alloying process by the energy driving force by hot energy supplied from the furnace. The purpose of this experiment is to observe the rapid plastic deformation effect to the sintering time, where

it is assumed that the rapid plastic deformation treatment will shorten the sintering time compared to the time consuming by the normal sintering.

THEORY

The simplest method to synthesize certain materials from powder elements components is powder metallurgy. In some cases the powder-metallurgy method does have its own advantages. Such as a more homogenous distribution of alloying elements, energy conservation and a comparative easiness in molding to match the geometrical shape of the dies. In short the synthesis of materials using the powder metallurgy technique is summarized as follows: first the composition of the alloying elements is determined based upon the design's requirements, next the required amount of each element is weighed using the digital balance; the second step is to blend the main components together with the alloying elements using the milling process until a homogenous blend is attained. The third step involves the making of pellets by compaction pressure method. In this case, the powder mixture was compacted into pellets. Afterwards, all pellets are sintered and followed by rapid plastic deformation process carried out by forging in order to enhance its Gibbs free-energy ^[1]. The rapid plastic deformation process is carried out for every half hour during the sintering, and then continued by homogenization at the same temperature as the sintering temperature. The example of plastic deformation process commonly used are forging and rolling; forging is a rapid plastic deformation process, meanwhile rolling is a slow plastic deformation process. The forging process brings about a massive damage to the crystal's structure. The crystal's area would shrink because of the thrust exerted by much dislocation of grain boundaries. The sharp

increase in number of dislocation formation around the grain boundary is causing the grain-boundary area to expand resulting in the widening of areas containing dislocations. Therefore the stacking fault energy, SFE, in this area also increases which is microscopically speaking and in a more general view could be considered to be an increase of the material's Gibbs free energy. The entire process starting from sintering with the aid of plastic deformation treatment and finished with homogenization takes only seven to eight hours and is comparatively less time-consuming than the available classical sintering process ^[1,-3]. Time preservation is made possible largely by the plastic deformation process which in turn enhances the free energy of the material, and therefore obliterates the need for high activation energy; the accumulative activation energy in the advanced phase formation decreases and the resulting effect is time conservation. The conventional and commonly accepted rule in sintering sets down that the sintering temperature should be set at two third the value of the melting point (in K) of that particular material. However in the making of this material a higher sintering temperature is applied, which is set at a

slightly lower value below the lowest commercially available austenites's melting point ^[4], and this choice is also related to time conservation. To put it bluntly, the rapid plastic deformation technique does not concern itself with the formation process, but rather concerns itself more with enhancing the material's free-energy. Therefore it should be obvious that time conservation means conservation of energy and the amount of inert gas used in both the sintering- and homogenization process, and these in turn will put a better emphasis on process to make maximum and efficiency.

MATERIALS AND METHODS

• Materials

In this work, the new alloy code named A-1, constitutes seven different elements, nickel, chrome, titanium, manganese, silicon, carbon each with a specified weight-percentage tabulated in Table-1. These alloying elements are entirely of powder quality, all commercially available in the market, especially from Merck Co. Fe and Cr with 99.0% purity; meanwhile Ni-, Si-, Mn-, C- and Ti powders each has a purity of 99.9%.

Table 1. Composition of material alloying elements in weight percent.

Elements	Ni	Cr	Si	Mn	C	Ti
wt. %	20.0	20.0	1.5	2.0	0.08 ≤	0.5

Instrumentation

In this work several equipments and instruments were used. These include spatula, light-resistant plastic container, ball-milling machine, dies, press-machine, and furnace. Ball milling machine is BIPs 395204 series, 1996. Quenching hardened NKL steel-

die in cylindrical form with an outside diameter of 7.0 cm, and an inside diameter of 2.0 cm was used. Press machine with a maximum pressure of 20000 psi, is a Carver model 3912 S/N: 40.000-LOG 10785. Furnace for sintering used is thermo-line model F47920-26-80. As well as forging equipment, the special cylindrical furnace was made and

used by Center for Metallurgy Research, LIPI. Microstructure observations are carried out using optical microscope of the type UFX-DX (Japan). Diffraction intensity pattern was obtained using the Shimadzu X-ray diffractometer XD-610. Microstructure was observed by optical microscope and by SEM equipped by EPMA to observe the elemental distribution in some small area.

Method

The elemental components are determined according to their weight-percentages using the digital micro-balance; all alloying materials are then blended together by constant stirring in a special-purpose plastic container; the process is continued by refined-stirring using the ball milling machine for 24 hours. Each sample is synthesized from a powder-mixture (of about five grams each) poured into a die of 10.0 cm outer diameter and 2.0 cm inner diameter. The compaction of the powder-mixture used pressure slightly above 8000 psi, holding time is set for ten minutes each [1]. The ompacting pressure is determined by a previous preliminary pressure experiment carried out

on several samples to obtain an optimum pressure magnitude yielding the best compaction results [1]. After compaction process the samples were pre-sintered at 1050°C for 24 hours, and then sintered at 1250°C for 6 hours in argon inert gas medium; and after half hour of sintering, the samples were plastic deformed by forging, and again repeating during the sintering process. The temperature of 1250°C is chosen because this value is very close to the lowest melting temperature which is at around of 1400°C [4]. The sintering processes, are carried out alternately with the plastics deformation treatments, a brief description is presented in the diagram of figure 1. The samples could now undergo several preparations in conformity with the type of test or experimental method to be carried out later. As an example, the preparation for optical-microscopy study is carried out in conformity with the standard of sample preparation [6]. The samples' microstructure is examined by using an optical-microscope and phase verification is accomplished by examining the x-ray diffraction pattern intensity.

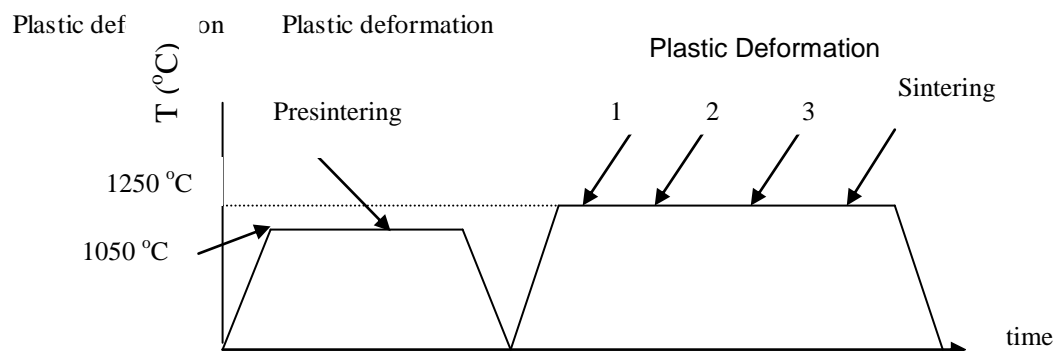


Fig.1. Pre-sintering and sintering process diagram interrupted by plastic deformation treatment. Arrows tagged with numbers [1], [2], and [3] are pointers to show some of the plastic deformation carried out during the sintering process. Pre-sintering was carried out at 1050°C, mean-while the sintering was carried out at 1250°C

RESULTS AND DISCUSSION

Based on the experimental data provided by the austenitic alloy synthesis experiments mentioned above a discussion and analysis of the data is now can be presented as follow. An optical microgram of the six hours long sintered sample is shown in Figure 2a. The microgram does confirm or provide evidence that in general the austenite phase has been attained, and the existence of irregularity area, formed grain boundary

large area so that the grain boundaries are like dispersed throughout the sample. This irregularity area is also evident from the presence of dislocations multiplication as the result of the several plastic deformation treatments on the sample during sintering at 1250°C for 6 hours escorted by rapid plastic deformation treatment. In the other side, as shown in Fig. 2a, in the big grain there are many sub-grains; it depicts that the sample also underwent re-crystallization process.

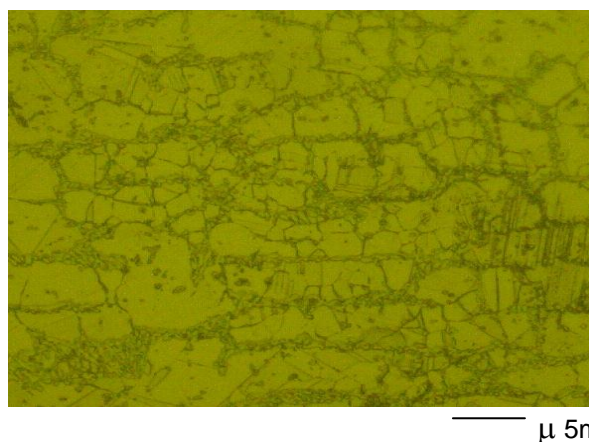


Fig. 2a. Optical microscope micrograph of the material (by magnification of 200x) as a result from sintering at 1250°C for 6 hours complemented by plastic deformation treatment.

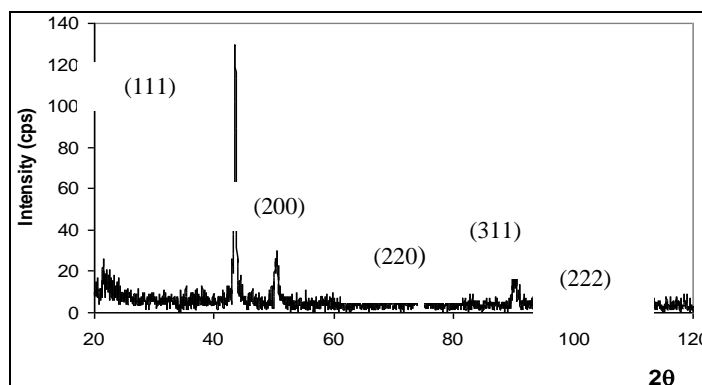


Fig. 2b. X-Ray diffraction pattern after sintering at 1250°C for 6 hours escorted by plastic deformation treatment in which the microstructure is shown in Fig. 2a. Notice the very prominent background intensity due to diffuse scattering confirming the sample's disordered structure caused by the plastic deformation treatment. This could be compared to the diffraction background intensity presented in Fig.-3b.

The grain boundary wide area can then be reduced by homogenization process, and after homogenization at 1250°C for 2 hours, the micrograph of the material is shown in Fig. 3a, where the width of grain boundary area is

reduced. If the homogenization process is continued up to above six hours, the grain boundary area is reduced to just a single line, as well as that in the usual form.

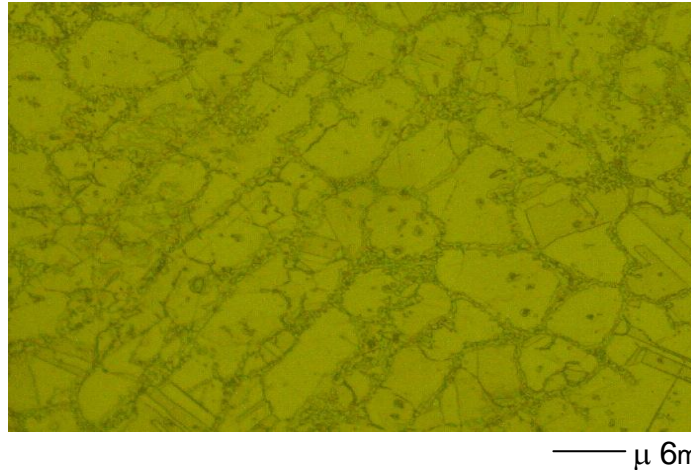


Fig. 3a. Optical microscope micrograph of the material (by magnification of 200x) as a result from sintering at 1250°C for 6 hours escorted by plastic deformation treatment and then homogenized at 1250°C for 2 hours.

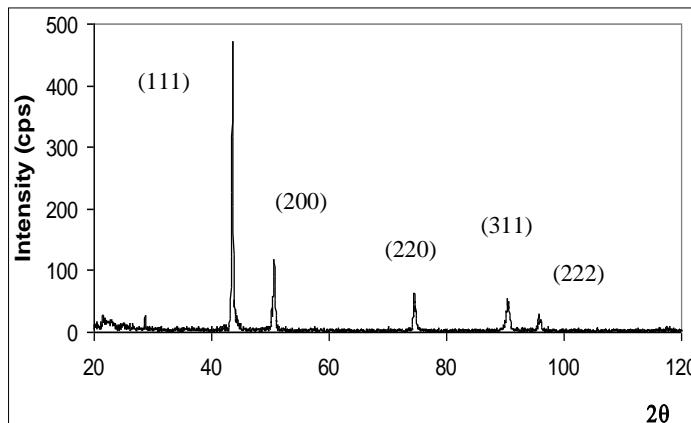


Fig. 3b. Diffraction pattern taken after sintering at 1250°C for 6 hours complemented by plastic deformation treatment plus homogenization at 1250°C for 2 hours, in Fig.3a. Here the background intensity is comparatively not as prominent as the one in figure-2b. This is caused by the disorder in the material, brought about by plastic deformation treatment is beginning to weaken due to homogenization process.

The X-ray diffraction pattern of the new sample is shown in Fig.-2b; the fcc crystallographic structure is clearly indicated by the diffraction peaks, which in turn points to a austenitic phase materials. The x-ray diffraction pattern of other samples of the same type also reveals an identical crystallographic structure. This result sort of increases the expectation that the new material has an austenite-type phase. By applying the indexing-method on the intensity pattern in Figures-2b, and also from analysis results using existing initial data by applying cubic structural model, the lattice parameter is calculated to be approximately 3.61 Å.

The optical micrograph of the sample taken after sintering at 1250°C for 6 hours escorted by plastic deformation treatment plus homogenization at 1250°C for 2 hours is shown in Fig.3a. From this micrograph appears that the grain boundary wide area was reduced after the same sintered sample homogenized at 1250°C for 2 hours compared to the micrograph of Fig.2a, in which the sintered sample is not homogenized yet. The grain-boundary area in Fig.3a is still wide, but the width of the disorder area is shrunk compared to the width of the disordered domain in Figure-2a, taken when the homogenization treatment on the

material has been running for two hours. The materials undergoing re-crystallization process for a longer period will have a lower Gibbs free energy in comparison to materials homogenized in shorter duration [3]. This is because during the homogenization process the number of dislocations will decrease since affected by advanced re-crystallization. In figure-3a the microgram also shows that re-crystallization occurs inside the larger grains, such that now smaller grains are enclosed inside the volume of relatively larger grains.

Diffraction pattern shown in Figure-3b is taken from the same sample with a microgram shown in Figure-3a. The diffraction peaks indicates that the material's crystal symmetry shows a singular face centered cubic structure. Therefore materials that follow a sequential cycle of sintering at 1250 °C for six hours, a plastic deformation treatment plus re-homogenization for two hours are in fact single-phase austenites. Here the background intensity is comparatively not as prominent as the one in Figure-2b. This is because the disorder in the material, brought about by plastic deformation treatment is beginning to weaken due to the homogenization process that reduced crystal disorder.

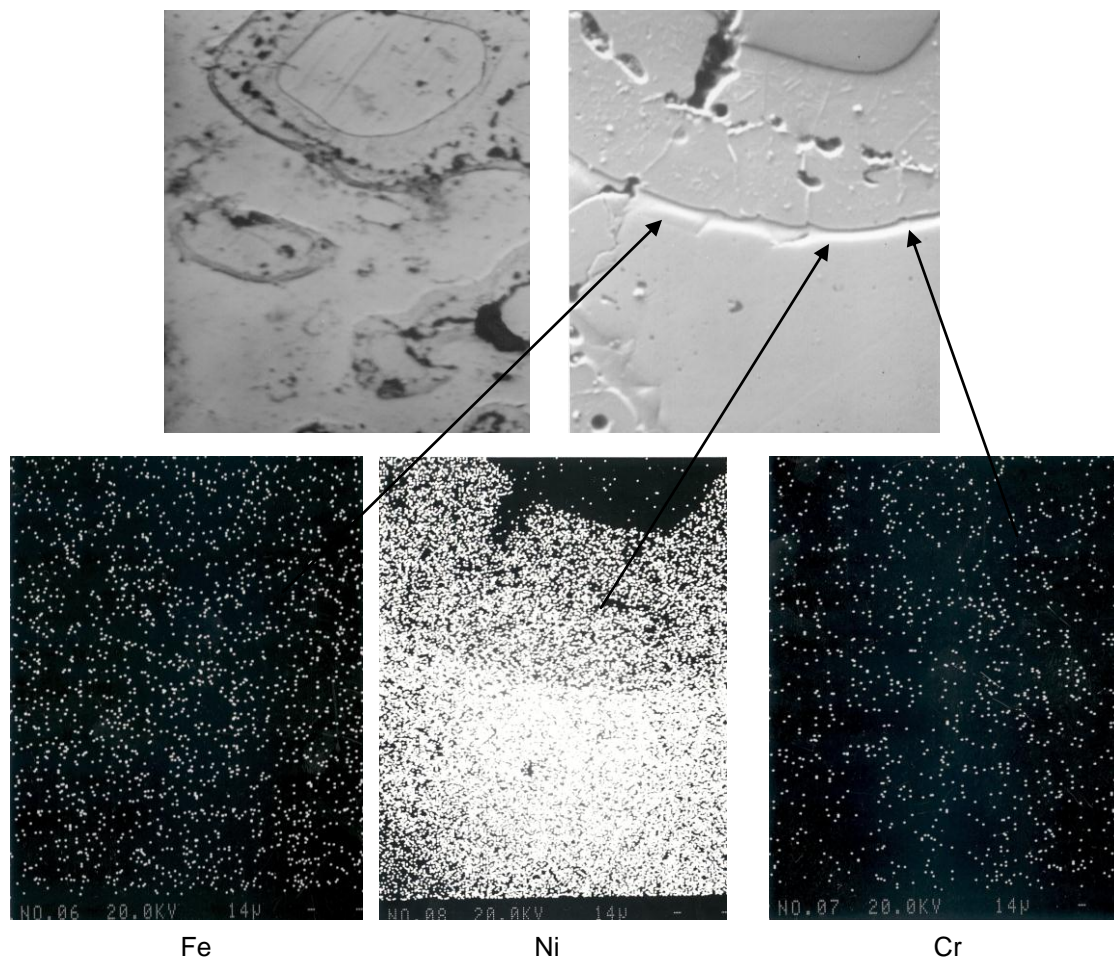


Fig. 4. Elements distribution of austenitic material (A1) after sintered for half hour (not plastic deformed yet).

The SEM micrograph of a small part of sintered sample at 1250°C for half hour is shown in Fig. 4, where the plastic deformation treatment has not been carried out yet. The micrograph is followed by the elemental mapping in the same area by EPMA, in which the sample major elements examined are iron, nickel, and chrome. So far, the examination of major elements by EPMA in

the SEM micrograph of Fig. 4, shows that the elements are not distributed yet homogeneously in which the sintering process is still conducted for half hour; in the spot area, distribution of iron elements are still relative small, as well as chrome elements. Inversely it appears that this spot area is dominated by nickel elements.

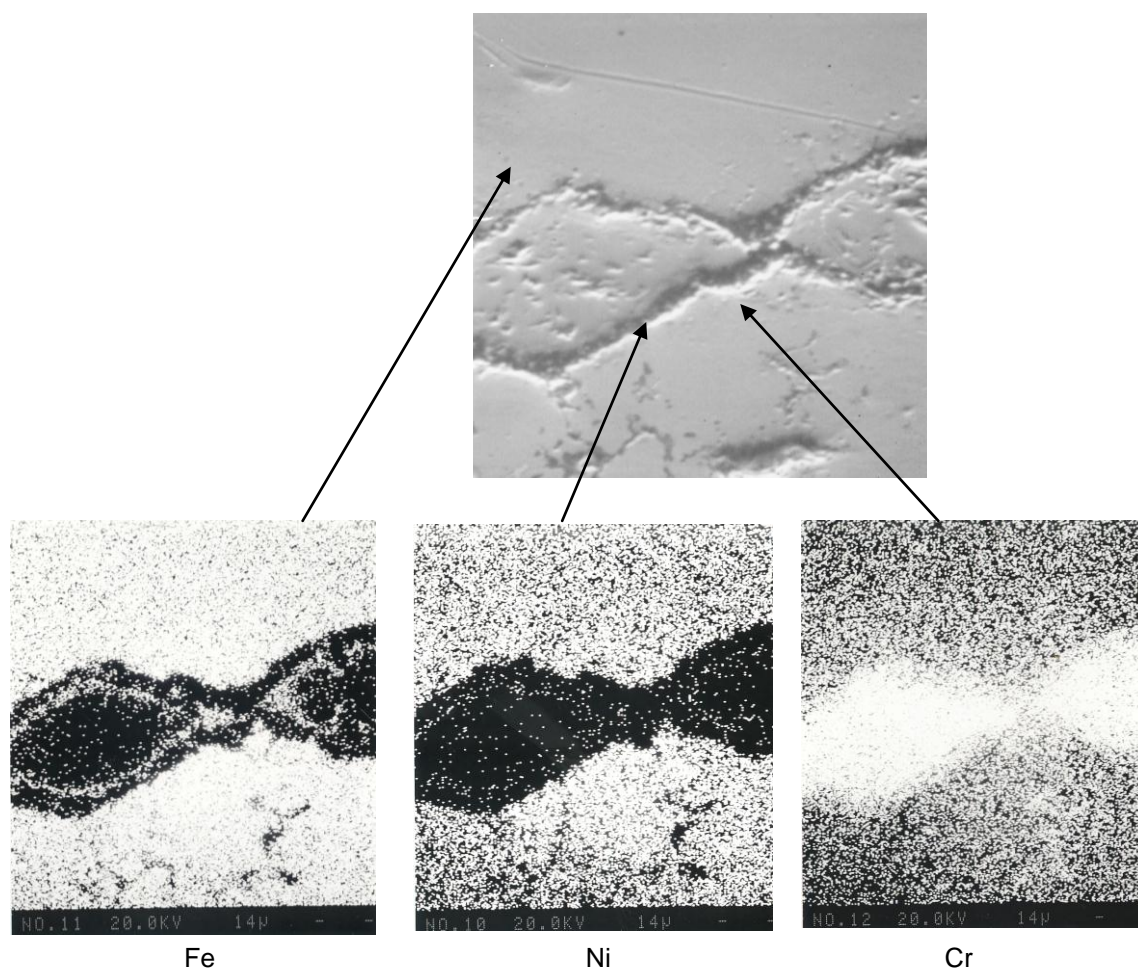


Fig. 5. Elements distribution of austenitic material (A1) after sintered by plastic Deformation treatment for 6 hours (not homogenized yet).

This examination major elements in the sample by EPMA is then continued to the sample sintered at 1250 ° C escorted by plastic deformation treatment for 6 hours and not homogenized yet, that depicted by the microstructure as shown in Fig. 2a, gives the result as well as SEM micrograph in Fig. 5. It seems that in the sample small part depicts by spot area of the micrograph of Fig. 5, the elements are relative distributed homogenously compared to that of Fig. 4. It appears also that in the spot in middle area position, the elements quantity are dominated by chrome; the second dominant quantity are iron Mean-while the nickel quantity is the third. But in overall, the quantity of the

element shows the material composition ratio as well as in the specification desired. So from the microstructure and also elemental distribution it can be said that the rapid plastic deformation treatment will reduce the sintering time compared to the normal sintering process ^[1, 3].

CONCLUSION

From the investigation and the discussion presented above it can be concluded that the rapid plastic deformation treatment during the sintering process will reduce the sintering time; the reason is the rapid plastic deformation treatment will rise

the dislocations multiplication that provide the disorder crystal structure; and the disorder crystal structure make the material in the meta-stable condition due to rise of the material's free-energy or potential energy, converted to added energy during the sintering process.

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